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TRIBOLOGICAL PROPERTIES OF STEEL/TiB_2 COMPOSITES PREPARED BY SPARK PLASMA SINTERING

WŁASNOŚCI TRIBOLOGICZNE KOMPOZYTÓW STAL/TiB_2 OTRZYMYWANYCH METODĄ SPS

The mechanical and tribological properties of sintered 316L stainless steel composites with TiB_2 submicroparticles were investigated. The composites were manufactured by Spark Plasma Sintering (SPS). The wear behaviour was studied by using a ball-on-disc wear tester at room temperature. The worn surface were analysed using Scanning Electron Microscopy (SEM). The results indicated that the friction coefficient and the wear resistance of composites with the same content of TiB_2 particles depend on the sintering conditions.

Keywords: ball-on-disc method, friction coefficient, wear rate, composites, Spark Plasma Sintering (SPS)

W prezentowanej pracy zbadano właściwości mechaniczne oraz tribologiczne materiałów kompozytowych umacnianych submikrocząstkami ceramiki TiB_2. Do wytworzenia spieków kompozytowych zastosowano plazmowe spiekanie iskrą elektryczną (SPS). Badania właściwości tribologicznych przeprowadzono w temperaturze pokojowej w układzie ball-on-disc. Powierzchnie próbek po testach ścierania obserwowano za pomocą skaningowej mikroskopii elektronowej. Uzyskane wyniki badań wykazały, że właściwości tribologiczne zależą od warunków spiekania dla kompozytów zawierających taką samą ilość ceramiki TiB_2.

1. Introduction

Titanium diboride (TiB2) has attracted much attention for various applications, such as cutting tools, wear-resistant parts, high-temperature structural materials and lightweight impact resistant armour material [1,2]. Also, TiB_2 has been identified as the most attractive reinforcement to steel matrix composites, mainly due to its low density, high melting temperature, low coefficient of thermal expansion (CTE), excellent wear and corrosion resistance, good wettability and good chemical compatibility with steel matrix [3-6]. Furthermore, TiB_2 is expected to be able to improve the wear resistance of steel due to its excellent mechanical properties like high hardness and high Young's modulus [7,8].

The friction coefficient and wear rate of composites depend on the type of matrix, reinforcement chemistry and volume, surface roughness, counterbody material and the experimental conditions, such as sliding speed, load and environment (humidity, atmosphere, etc.). The effect of particles additions on the tribological performance of composites is complicated. Incorporation of a ceramic phase to steel matrix can result in improved wear performance [9,10]. Ashok and Karabi [11] showed that the abrasive wear resistance and the coefficient of friction of unreinforced austenitic steel, and TiC and (Ti,W)C-reinforced composites decrease as the load increases. The TiC and (Ti,W)C-reinforced composites show better dry sliding wear resistance than that of unreinforced austenitic steel matrix. The abrasive wear resistance of (Ti,W)C-reinforced composite is higher than that of TiC-reinforced composite. Lin and Xiong [12] fabricated the 316L stainless steel composites with various fractions of TiC particles using warm compaction and microwave sintering. Their analysis showed that the volume loss of wear tests initially decreases with increasing TiC content up to 5 wt%, then it slightly increases as the TiC particles content increases to 10 and 15 wt%. The composites with 5 wt% TiC addition offer a high wear resistance. Velasco *et al.* [13] manufactured composites using 316L austenitic stainless steel as matrix and TiAl intermetallic as reinforcement. The effect of intermetallic particles on wear behaviour of steel was assessed by a complete tribological study using a pin on disk method. The wear behaviour of 316L austenitic stainless steel is improved when TiAl intermetallic reinforcements are added and sintered at 1250°C. Friction coefficient is only slightly affected by sintering temperature. Vardavoulias *et al.* [14] investigated the dry sliding wear behaviour of two austenitic stainless steels (304L and 316L) and their composites containing two different types of ceramic particles (Al_2O_3 and Y_2O_3). Additionally, two different sintering activators (BN and B_2Cr) were used. The investigations indicate, that the presence of ceramic particles (Al_2O_3 and Y_2O_3) and sintering activators (BN and B_2Cr) was found to improve wear resistance. The best properties were obtained for the combination of Al_2O_3 and B_2Cr, since the ceramic particles could limit plastic deformation while sintering activators decreased final porosity.

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Several studies [15-19] have been reported to investigate the wear behaviour of composites with TiB_2 particles. Sulima *et.al* [15] showed that the friction coefficient of the steel composites decreases with increasing TiB_2 content. The best tribological properties were obtained for the austenitic stainless steel reinforced with 20 vol.% TiB_2 particles. However, Tjong *et al*. [16,19] studied the properties of the composites reinforced with various volume fractions of TiB_2 particles. They indicated that the addition of TiB_2 particles was very effective to improve the wear resistance and ductility of austenitic stainless steel.

The aim of the study was investigation of the tribological behaviour of the composites with various content of TiB_2 submicroparticles. The tribological properties were studied using a ball-on-disk tester.

2. Experimental procedure

The TiB_2 powder (2.5-3.5 in average size, 99.9 wt.% in purity, H.C. Starck) and commercial AISI 316L austenitic stainless steel (25 μm in average size, KAMB Import-Export) were used in the present study. The chemical composition of the austenitic stainless steel powder was 17.20 wt% Cr, 12.32 wt% Ni, 2.02 wt% Mo, 0.43 wt% Mn, 0.89 wt% Si, 0.03 wt% S, 0.028 wt% P, 0.03 wt% C and balance of Fe. The following compositions were investigated:

- Steel AISI 316L + 2 vol.% TiB_2
- Steel AISI 316L + 4 vol.% TiB_2
- Steel AISI 316L + 6 vol.% TiB_2
- Steel AISI 316L + 8 vol.% TiB_2.

For comparison, powder of commercial AISI 316L austenitic stainless steel was sintered.

The samples were obtained by powder metallurgy route. The SPS machine type HPD 5 (FCT System, Germany) was used in this study. Materials were compacted in a graphite die with 20 mm inner diameter, using a maximum pressure of 35 MPa at vacuum. Maximum pressure was obtained after 10 min of test duration. Vacuum and pressing time of 10 minutes was to vent the mixture. After that, the SPS furnace chamber introduced argon, which acted as a protective gas and the sintering process was carried out. The powders were sintered at temperature of 1000°C and 1100°C, for duration of 5 and 30 min. The heating rate was 200°/min.

After sintering, the relative density of composites was measured by Archimedes water immersion method. In this technique, density is determined by measuring the difference between the specimen's weight in air and when it was suspended in distilled water at room temperature. The Vickers's microhardness of the composites was determined at 2.94 N load using a FM-7 microhardness tester.

Tribological tests were performed using a ball-on-disc wear machine UMT-2T (producer CETR, USA). The experimental procedure followed the ISO 20808:2004(E) [20,21]. For ball-on-disk method the sliding contact is brought by pushing a ball on a rotating disc specimen under a constant load. The loading mechanism applies a controlled load F_n to the ball holder. The friction force was measured continuously during the test using the extensometer. For each test a new ball is used. Specimens were washed in high purity acetone and dried. After the ball and sample were mounted, materials are washed in ethyl alcohol and then dried. The wear test conditions were:

- ball made of Al_2O_3, diameter of 3.175 mm,
- friction track diameter: 4 mm,
- sliding speed: 0.1 m/s,
- total sliding distance: 200 m,
- test duration: 2000s,
- load applied: 4 N,
- room temperature.

The values of friction coefficient were calculated from the following equation:

$$\mu = \frac{F_f}{F_n L} \tag{1}$$

where F_f is the measured friction force, and F_n is the applied normal force, L– sliding distance [m].

Following the wear test, the specific wear rate was calculated. For the wear track on the disc specimen, the cross-sectional profile of the wear track at four places at intervals of 90°using a contact stylus profilometer was measured with accuracy of measurement in the vertical axis of 0.01 μm, in the horizontal axis of 0.1 μm. The cross-sectional area of the wear track was calculated using a custom-developed software. Specific wear rate according to wear volume was calculated by means of equation:

$$W_{V(disc)} = \frac{V_{disc}}{F_n L} \tag{2}$$

where: $W_{V(disc)}$– specific wear rate of disc [mm^3/Nm]; V_{disc}– wear volume of disc specimen [mm^3]; F_n– applied load [N]; L– sliding distance [m].

Next, the specific wear rate of Al_2O_3 ball was calculated from the difference due to wear between the mass of the ball specimen before and after test, by means of equation:

$$W_{s(disc)} = \frac{m_{before} - m_{after}}{F_n L \rho_{ball}} \tag{3}$$

where: $W_{s(disc)}$– specific wear rate of Al_2O_3 ball [mm^3/Nm]; m_{before}– the mass before test [kg]; m_{after}– the mass after test [kg]; F_n– applied load [N], ρ_{ball}– density of Al_2O_3 ball [kg/m^3].

For morphological characterization of the composites Olympus GX-51 optical light microscopy was used. The microstructure of composites after tribological tests was evaluated by scanning electron microscope (SEM) JEOL JSM 6610LV.

3. Results and discussion

The relative density values of the sintered composites for different sintering conditions are shown in Figure 1. It can be seen that the densification increases with the sintering temperature, reaching maximum for the composites obtained at 1100°C. For these composites a maximum relative density of 98-99% was obtained. However, the increase of duration of test to 30 minutes at the same temperature does not significantly affect the improvement of the density. It follows that, the densification processes of composites occurred most intensively directly during the first minutes of SPS process. In the first few minutes, the appropriate density was obtained.

The application of longer time does not significantly modify density.

The optical micrographs of the 316L austenitic stainless steel and selected composites with 8 vol.% TiB_2 prepared by SPS process are shown in Figure 2. Generally, TiB_2 particles are homogeneously distributed in the steel matrix (Fig. 2b,c). The application of the temperature of 1100°C changed microstructure of the composites. The formation of a few fine phases in steel grains was observed (Fig. 2c). The details of the microstructure and mechanism of the formation of new phases will be discussed elsewhere [22].

Figure 3 shows the relation between microhardness and sintering conditions. It can be seen that the microhardness of the composites increases with the temperature. A maximum microhardness was obtained for composites sintered at 1100°C for 30 minutes. The composites with 2 vol.%, 4 vol.%, 6 vol.% and 8 vol.% TiB_2 reached microhardness HV0.3 of 330, 385, 405 and 436, respectively.

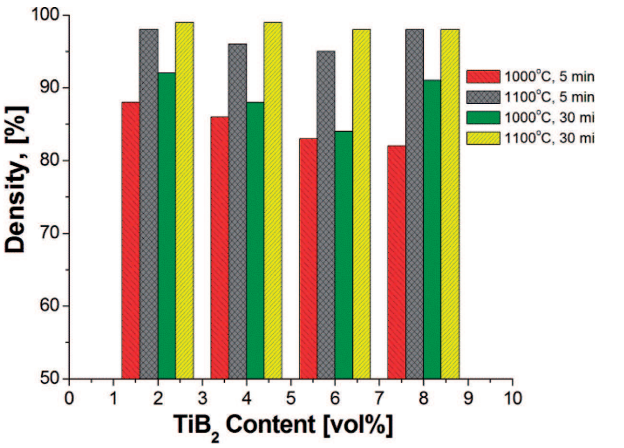
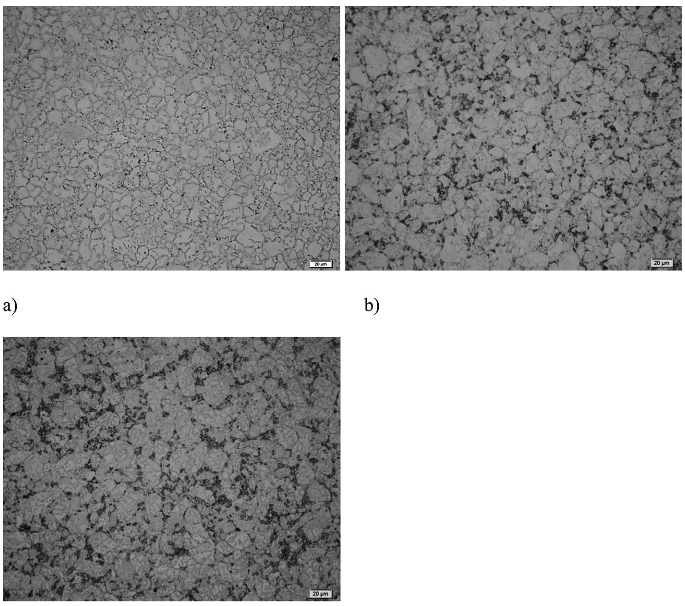


Fig. 1. The relative density of studied composite materials



a) b) c)

Fig. 2. Selected micrographs before tribological tests of a) 316L steel (1100°C, 30 min) and b) composite with 8 vol.% of TiB_2 (1000°C, 5 min) and c) composite with 8 vol.% of TiB_2, (1100°C, 30 min)

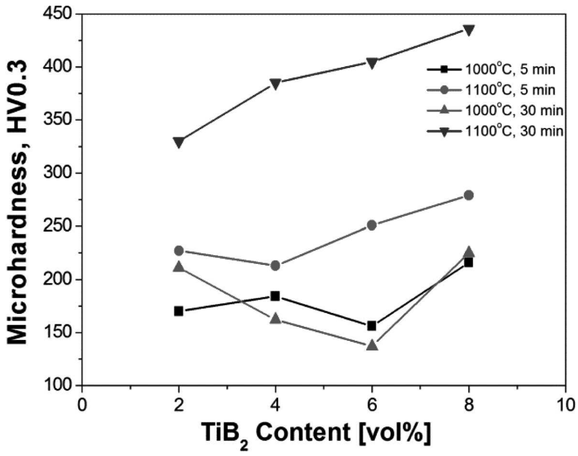


Fig. 3. The hardness of composites prepared at different sintering conditions

The wear resistance of the composites was evaluated by measuring the friction coefficient and wear rate using ball-on-disk method. Typical variation curve of the friction coefficient of the sintered materials with an applied load of 4 N and a sliding speed 0.1 m/s is given in Figure 4. The addition of TiB_2 reduced essentially the friction coefficient of the composites. The friction coefficient is the highest for the composites with 2 vol.% TiB_2. Next, the friction coefficient decreases gradually reaching the lowest values for composites with 8 vol.% TiB_2. The TiB_2 additions cause the improvement of the wear resistance of austenitic stainless steel due to the high hardness of TiB_2 particles (3400 HV [23]) and the slow wear of reinforcement. It is interesting that the sintering conditions influence significantly the wear behavior of the composites. In the case of austenitic steel, the friction coefficient does not depend on the sintering conditions. Friction curves are similar for the steel which were sintered at different conditions of sintering. The values of the friction coefficient are very similar in the range of 0.60-0.62. The sintering conditions have made remarkable role in improving the tribological behaviour of sintered composites (Fig.5). It can be found that the friction coefficient of composites with the same content of TiB_2 particles depends on phase and microstructural composition of the steel matrix and microstructure depend on the sintering conditions. It was observed that the dependence of friction coefficient on temperature and time shows a similar trend for all composites. The application of higher sintering temperature decreases the friction coefficient of composites with the same content of TiB_2. In the case of composites with 8%vol. TiB_2, the friction coefficient is 0.51 for sintering temperature of 1000°C (duration of 5 minutes) and reduces to 0.38 at sintering temperature of 1100°C (duration of 5 minutes). Likewise, the duration of the process has an effect on the values of friction coefficient of composites. It is observed that the friction coefficient reduce with increasing of duration. It can be seen for composites with 6% vol. TiB_2, for which the friction coefficients are 0.43 and 0.36 for 5 and 30 minutes (1100°C), respectively.

Figure 6 shows the variation of the specific wear rate of composites obtained using ball-on-disk method under applied load of 4N. It is clear from this figure that the wear rate decreases with the increase TiB_2 content. The wear rate of the unreinforced steel is higher than that of the composites which were sintered in different conditions. The lower wear rates of composites with higher amount of TiB_2 particles can be attributed to the higher hardness of composites. This is consistent with investigations Tjong and Lau [19] which indicated that the hardness of the composites tends to increase with increasing TiB_2 content, thus the abrasive wear resistance of the composites improves considerably with increasing TiB_2 content.

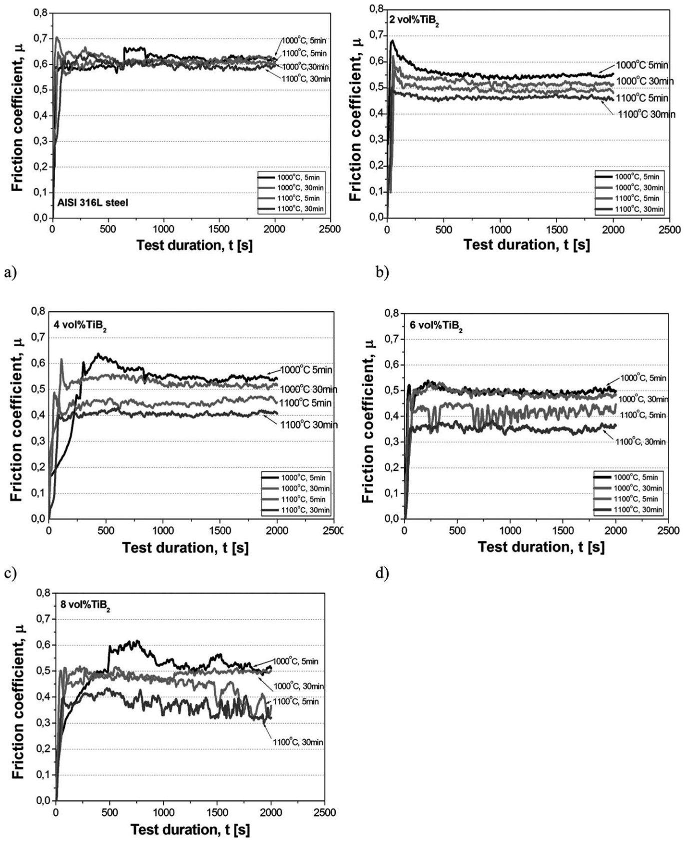


Fig. 4. Typical COF curves of the materials with testing time for austenitic steel and composites reinforced with of TiB_2 particles (a-e)

Also in the case of tested materials, the homogeneous distribution of the fine reinforcements is favorable for the improvement of the wear resistance. The next factor can be good interfacial bonding in composites obtained at 1100°C. This materials are characterized by the highest level of consolidation. It is important that the applied sintering conditions also influence the specific wear rate of composites. The investigations indicate that the specific wear rate improves with increase of the sintering temperature. For the sintering temperature of 1000°C and duration of 5 and 30 minutes the material demonstrates the higher wear rates. This results suggest the relationship between density and specific wear rate of composites. It was observed that for a higher degree of consolidation the lower wear rate was achieved. Higher loss of material will occur in case of less dense samples due to the loss of adherence of particles that can lead to higher wear rate. The smallest values of wear rate ($208 \cdot 10^{-6}$ mm^3/Nm) have composites with 8 vol.% of TiB_2.

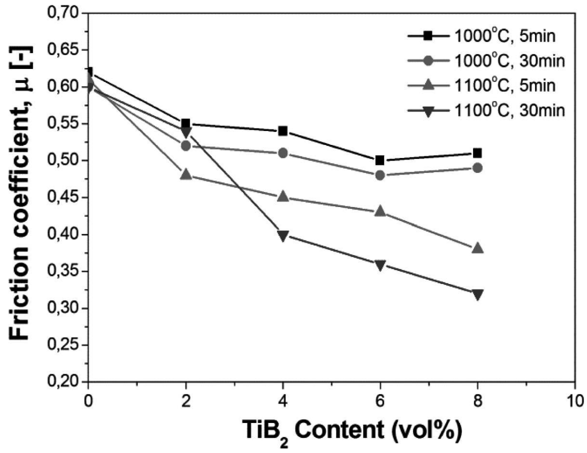


Fig. 5. Variation of friction coefficient with TiB_2 content for sintered composites

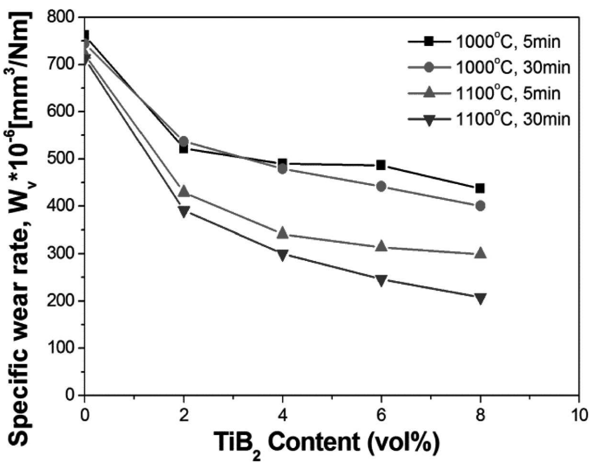


Fig. 6. Variation of specific wear rate with TiB_2 content for sintered composites, measured using ball-on-disc method

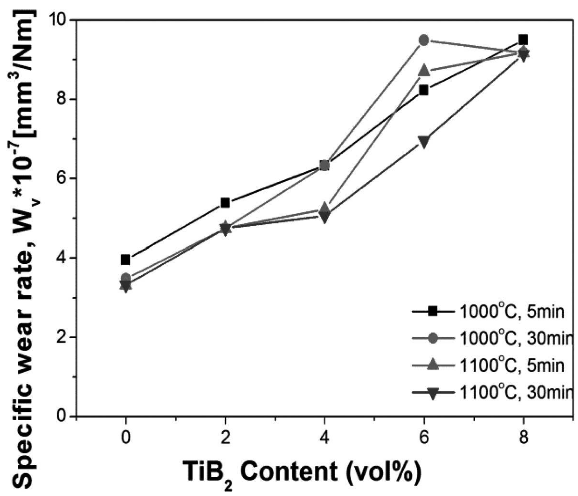
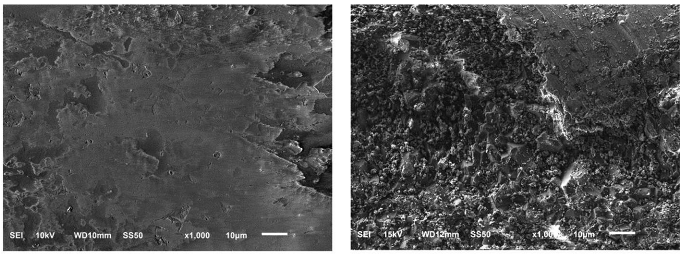


Fig. 7. Variation of specific wear rate of Al_2O_3 balls

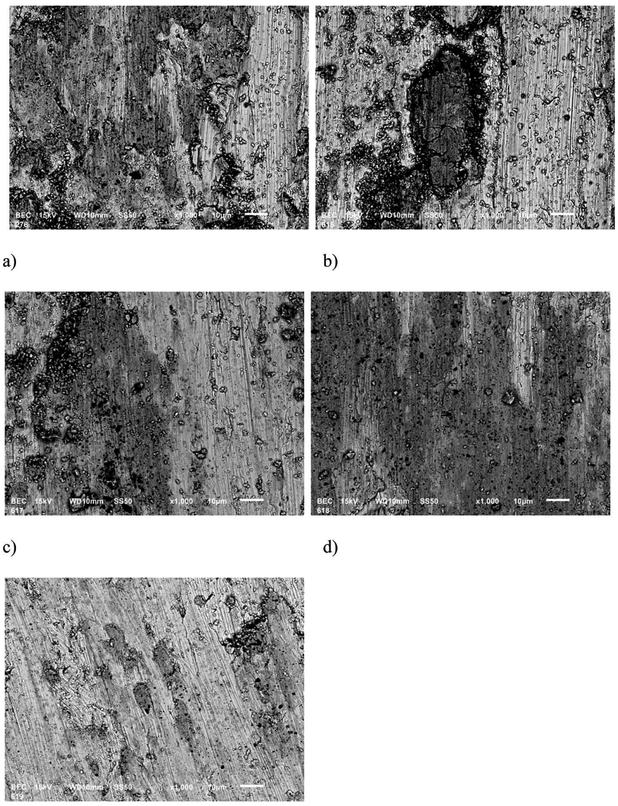
Also, the analysis of weight loss of Al_2O_3 balls which were used in the ball-on-disk tests was performed. It was indicated that the wear rate of balls depends on the content of strengthening phase in a composite and sintering conditions (Fig. 7). The increase of specific wear rate of Al_2O_3 ball with increasing TiB_2 content in steel matrix was observed. It is result of the presence of hard ceramic particles (3400 HV [23]) mainly, which effectively reinforce the matrix and protect it from serious abrasion. For comparison, the hardness of Al_2O_3 ball is 2000 HV [23]. Additionally, it was showed that the application of the higher sintering temperature contributed to a decrease of wear rate of composites. The lower wear rate can be attributed to increase of the hardness.



a) b)

Fig. 8. SEM micrograph of the surface of Al_2O_3 ball: a) before and b) after tribological test

Figure 8 presents microstructure of the surface of Al_2O_3 ball before and after tribological test. After each test a worn surface of the ball was damaged and coarse (Fig. 8b), with clearly visible places where Al_2O_3 particles were taken out from the surface of the ball. This indicates that the process of wear of the Al_2O_3 ball occurs during contact with the surface of the composite. Figure 9 a-d shows the worn surfaces of the austenitic steel and composites reinforced TiB_2 particles, which were sintered at temperature of 1100°C for 30 min. Wear is abrasive in all tested materials. Plastic deformation with characteristic grooves was observed. The worn surface of this composites is characterized by fine scratches to distinct grooves. Increase of the TiB_2 content resulted in a decrease in the plastic deformation of the composites. It can be seen from the SEM micrographs that the worn surfaces of the composite materials (especially with 6 and 8% vol. TiB_2) exhibit somewhat less plastic ploughing and cutting compared to that of the AISI 316L austenitic steel. This may be due to the presence of hard TiB_2 reinforcements on the worn surfaces of the composite materials.



e)

Fig. 9. SEM micrograph of the worn surface of composites with: a) 316L steel, b) 2 vol.% of TiB_2, c) 4 vol.% of TiB_2, d) 6 vol.% of TiB_2 and e) 8 vol.% of TiB_2 which were sintered at temperature of 1100°C for 30 min

4. Conclusions

The wear behavior of composites reinforced with TiB_2 was studied using a ball-on-disc method. These materials were obtained by Spark Plasma Sintering. The increase in TiB_2 content improves the wear resistance of composites. The SPS conditions have significant effect on tribological properties of the composites. Friction coefficient is affected by sintering temperature. The application of higher sintering temperature decreases the friction coefficient of composites with the same content of TiB_2. It was observed, the reduction of wear rate with increasing the temperature and duration of sintering. The best value of friction coefficient (0.32) and specific wear rate ($208 \cdot 10^{-6}$ mm^3/Nm) have composites with 8% vol. TiB_2 which were obtained at the temperature of 1300°C for 30 minutes.

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